NASA-CR-54281

STUDIES OF ALKALI METAL CORROSION ON MATERIALS FOR ADVANCED SPACE POWER SYSTEMS

Quarterly Progress Report No. 2
For Quarter Ending December 26, 1964

By
R.W. HARRISON

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
CONTRACT NAS 3-6012

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC
CINCINNATI, OHIO 45215

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STUDIES OF ALKALI METAL CORROSION ON MATERIALS FOR ADVANCED SPACE POWER SYSTEMS

QUARTERLY PROGRESS REPORT 2

Covering the Period

September 26, 1964 to December 26, 1964

Written by

R. W. Harrison

Approved by
J. W. Semmel, Jr.
Manager, Materials and Processes

Prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-6012

Technical Management
NASA - Lewis Research Center
Mr. R. L. Davies

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

CONTENTS

TABLES

ILLUSTRATIONS

ILLUSTRATIONS (Cont'd)

I. INTRODUCTION

The program reviewed in this second quarterly progress report, covering from September 26, 1964 to December 26, 1964, is sponsored by the National Aeronautics and Space Administration. Its purpose is to examine the influence of stress on the corrosion behavior of an advanced refractory alloy in potassium (Task I) and to investigate corrosion mass transfer effects in a stainless steel-columbium alloy-potassium system (Task II).

Task I

While there is considerable evidence that refractory alloys have excellent corrosion resistance to potassium, there are few experiments which describe the possible effects of stress on corrosion when the stress is sufficiently large to produce substantial amounts of creep during the test. It is appropriate for comparative purposes to study an advanced refractory alloy which has demonstrated excellent corrosion resistance to refluxing potassium during long-time exposures conducted at relatively low stresses at 2000°F. In this regard, D-43 columbium base alloy, in the form of welded capsules, has been tested in potassium under refluxing conditions for periods of 5,000 and 10,000 hours at temperatures on the order of 2000°F (Ref. 1) and has been selected for inclusion in this program.

The D-43 alloy reflux capsules shall be tested under conditions which result in about 5 to 10% strain during a 500- to 2,000-hour exposure period in the 2000° to 2200°F temperature range. The reflux capsules used in this study will be of similar size to those previously described (Ref. 1). The capsule wall shall be reduced in the potassium liquid region and in the vapor condensing region to provide gauge sections where the extent of creep can be measured. Moderate temperature adjustments can be made during the test to achieve the desired strain-time conditions.

Task II

The use of stainless steel, rather than refractory alloys, for power plant radiator construction and for the lower temperature portion of experimental facilities constitutes material and fabrication cost savings. Two methods of employing this approach are: use of co-extruded, stainless steel shell-refractory alloy core, tubing in the radiator or use of an all stainless steel radiator joined to the system by a bimetallic joint. Although the latter approach is preferred considering cost and problems associated with fabrication and joining of co-extruded tubing, a major uncertainty and limitation arises from the mass transfer of interstitial elements from the stainless steel to the refractory alloys through the alkali metal.

It is well established that the carbon and nitrogen transfer from Type 316SS to Cb-1Zr alloy at temperatures near 1500°F (Ref. 2). While some important aspects of this mass transfer behavior have been examined, several critical details require additional investigation. There is a need to define acceptable time and temperature conditions of operation in terms of maintaining satisfactory performance of the refractory alloys, such as Cb-1Zr alloy. Also, there are certain metallurgical aspects of this behavior which should be investigated in an effort to eliminate or reduce the mass transfer rate. In the latter category, it is most appropriate to consider the stabilization of carbon and nitrogen in the stainless steel by the addition of metallic elements which form carbides and nitrides of high thermodynamic stability. Commercially available, titanium stabilized, Type 321SS is one such alloy. A comparative investigation of this alloy and Type 316SS should indicate the ability of the titanium addition to reduce or eliminate interstitial mass transfer in a stainless steel-Cb-1Zr alloy bimetallic system. Columbium-1% zirconium alloy specimens will be exposed to liquid potassium in Type 321SS and Type 316SS capsules for 1,000 hours at 1400°F under isothermal conditions to evaluate this premise.

II. SUMMARY

During the second quarter of this program, the topics abstracted below were covered. The results are interpretatively presented in this report.

Task I - Stress Corrosion Reflux Capsule Tests

The D-43 alloy bar for the fabrication of the reflux capsules was received and evaluated. Measured creep strengths were higher than those previously reported from the data compilation.

The stress corrosion reflux capsule design was completed; engineering drawings were approved by the NASA Technical Manager. Three capsules have been machined and received.

With the exception of the LVDT's and the alumina probes, all supporting components for the reflux capsule test facility have been machined and/or are on hand.

A boiling nucleator was designed and is to be employed in the capsule tests in order to avoid possible deformation in the reduced wall and/or fracture of the alumina probes as a result of boiling instabilities.

Task II - Bimetallic Isothermal Capsule Tests

Two Type 321SS and two Type 316SS isothermal capsules, each containing Cb-1Zr alloy sheet specimens, were filled with potassium and placed on test at 1400°F December 18, 1964. The planned test duration is 1,000 hours.

III. TASK I - STRESS CORROSION REFLUX CAPSULE TESTS

A. Material Procurement

Six pieces of 1.66-inch diameter x approximately 13-inch lengths of D-43 alloy bar stock were received. The bar was produced from a 8-inch diameter, double vacuum arc melted ingot (heat 42-478-02) which was double extruded at 2500°F. In each case a 4:1 extrusion ratio was employed. The resulting bar was machined and subsequently given a recrystallization anneal at 2600°F. Chemical analyses of samples taken from the bars are shown in Table I. Although fully recrystallized material, with a grain size of ASTM 6-8, was ordered, metallographic examination of transverse sections of the bars revealed the structure of the center core area to contain considerable amounts of residual cold work, Figure 1. However, since the core material would be removed in the fabrication of the reflux corrosion capsules and the remaining material around the periphery did meet the grain size requirements, Figure 2, fabrication proceeded as planned.

B. Capsule Design and Fabrication

The completed D-43 alloy reflux corrosion capsule design is shown in Figure 3. A minimum 0.020-inch thick reduced wall section was selected on the basis of available creep data on the D-43 alloy (Ref. 3). To validate the capsule design, additional creep data were obtained from specimens, Figure 4, machined from the D-43 alloy bar used in the fabrication of the actual capsules. These data are compared with the forementioned data (Ref. 3) in Figure 5. It should be noted that the test specimens were machined from the periphery of the D-43 alloy bar such that the gauge section of each specimen had a similar metallurgical morphology as the capsule reduced wall section.

The creep specimens were heated by a tantalum foil resistance element in a cold wall vacuum facility at 1×10^{-6} torr. Specimen elongation was measured by an external LVDT extensiometer system that was attached to the load train. The quality of the test environment was evaluated by a comparison of the post-test chemical analysis of a creep specimen tested for 134 hours at 2400°F with the pre-test analysis, Table II. The small oxygen increase shown would have little effect on the creep behavior of this specimen which represents the extreme conditions of time and temperature of all the specimens tested. In addition, the maximum time observed for 5% strain, the value of main interest in this study, was 50 hours.

TABLE I

CHEMICAL ANALYSES OF THE 1.66-INCH DIAMETER D-43 ALLOY BAR

(Heat 43-478-02)

Element	Vendor's Analyses ppm	GE Analyses ppm
Oxygen	56	48 (1)
Hydrogen	8	5 (1)
Carbon	865	--
Nitrogen	35	23 (1)
Tungsten	10.1 $^{w}/_{o}$	--
Zirconium	1.0 $^{w}/_{o}$	--
Columbium	Bal	--

(1) By Vacuum Fusion Techniques.

Figure 1. Structure of the Center Core From 1.66-Inch Diameter D-43 Alloy Bar Showing Evidence of Prior Working. (K5263)

Etchant: 20%HF-20%HNO_3-60%H_2O Mag: 100X

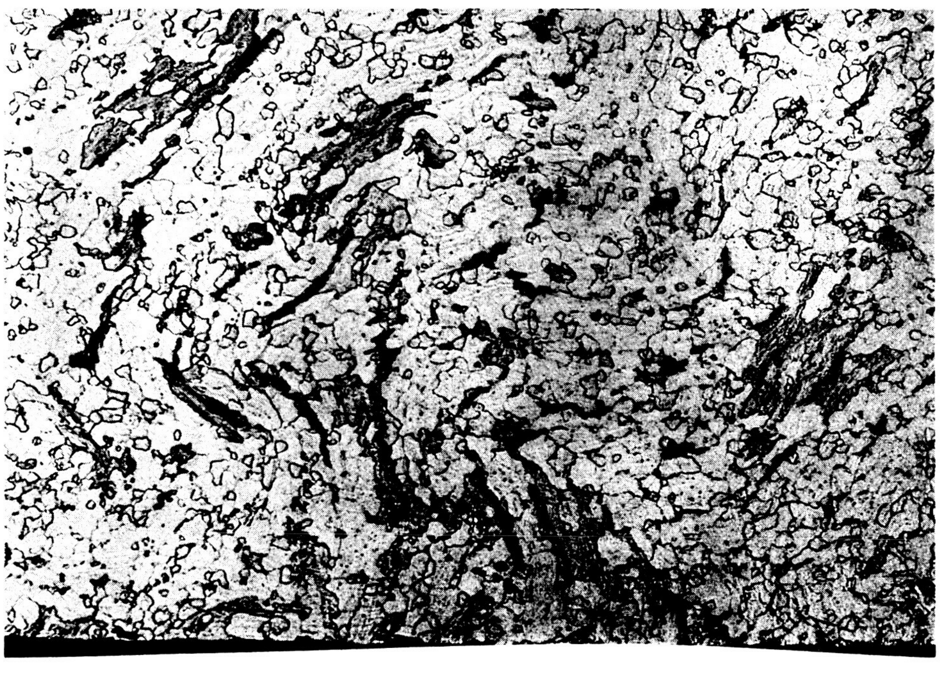
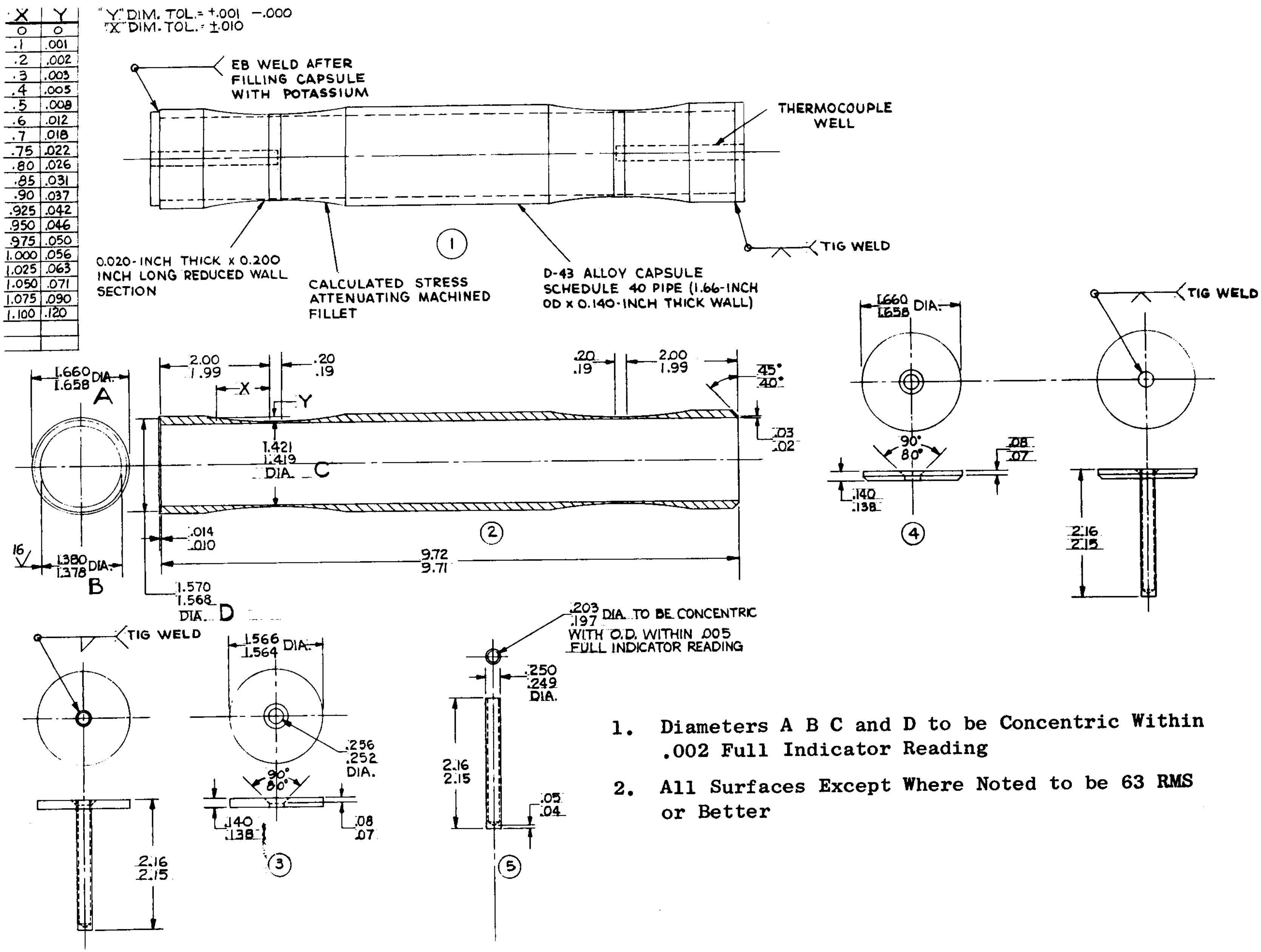


Figure 2. Recrystallized Structure of Outer Region of 1.66-Inch Diameter D-43 Alloy Bar. (K5264)

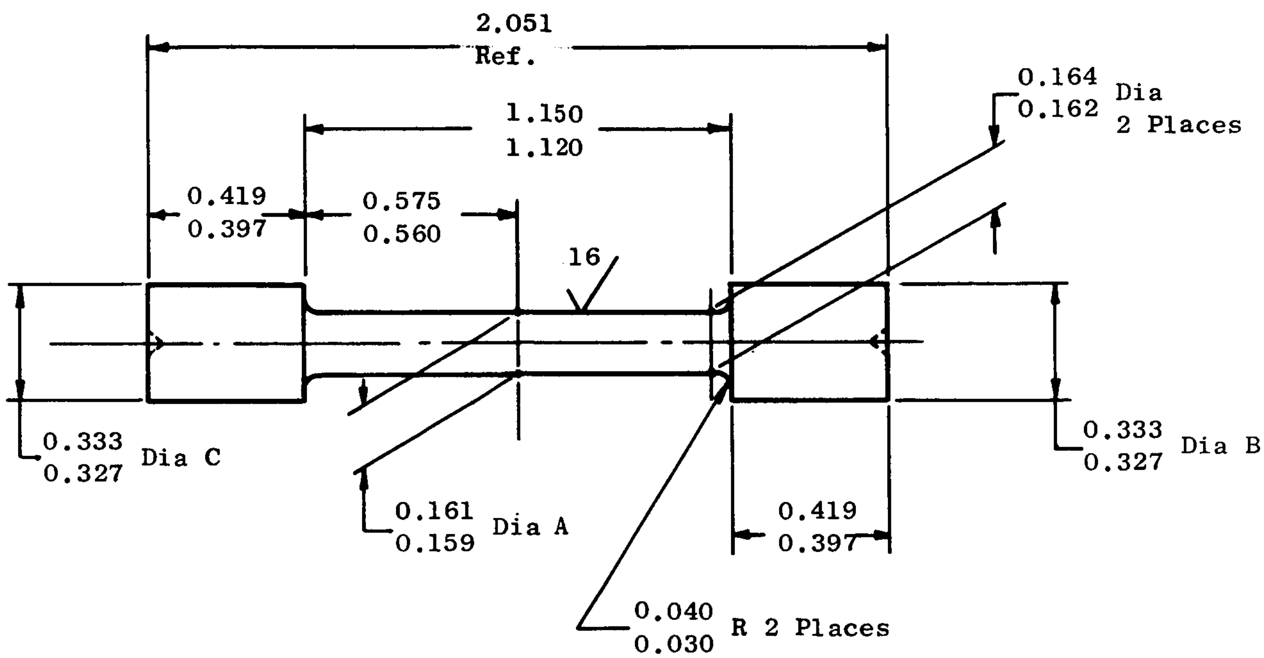
Etchant: 20%HF-20%HNO_3-60%H_2O Mag: 100X



X	Y
0	0
.1	.001
.2	.002
.3	.003
.4	.005
.5	.008
.6	.012
.7	.018
.75	.022
.80	.026
.85	.031
.90	.037
.925	.042
.950	.046
.975	.050
1.000	.056
1.025	.063
1.050	.071
1.075	.090
1.100	.120

1. Diameters A B C and D to be Concentric Within .002 Full Indicator Reading
2. All Surfaces Except Where Noted to be 63 RMS or Better

Figure 3. D-43 Alloy Reflux Corrosion Capsule Design with Reduced Wall Sections for Measuring Induced Strain.



Dia's B & C to be Concentric With Dia A Within 0.001 F.I.R.

Figure 4. Test Specimen Used for Creep Evaluation of D-43 Alloy in Vacuum.

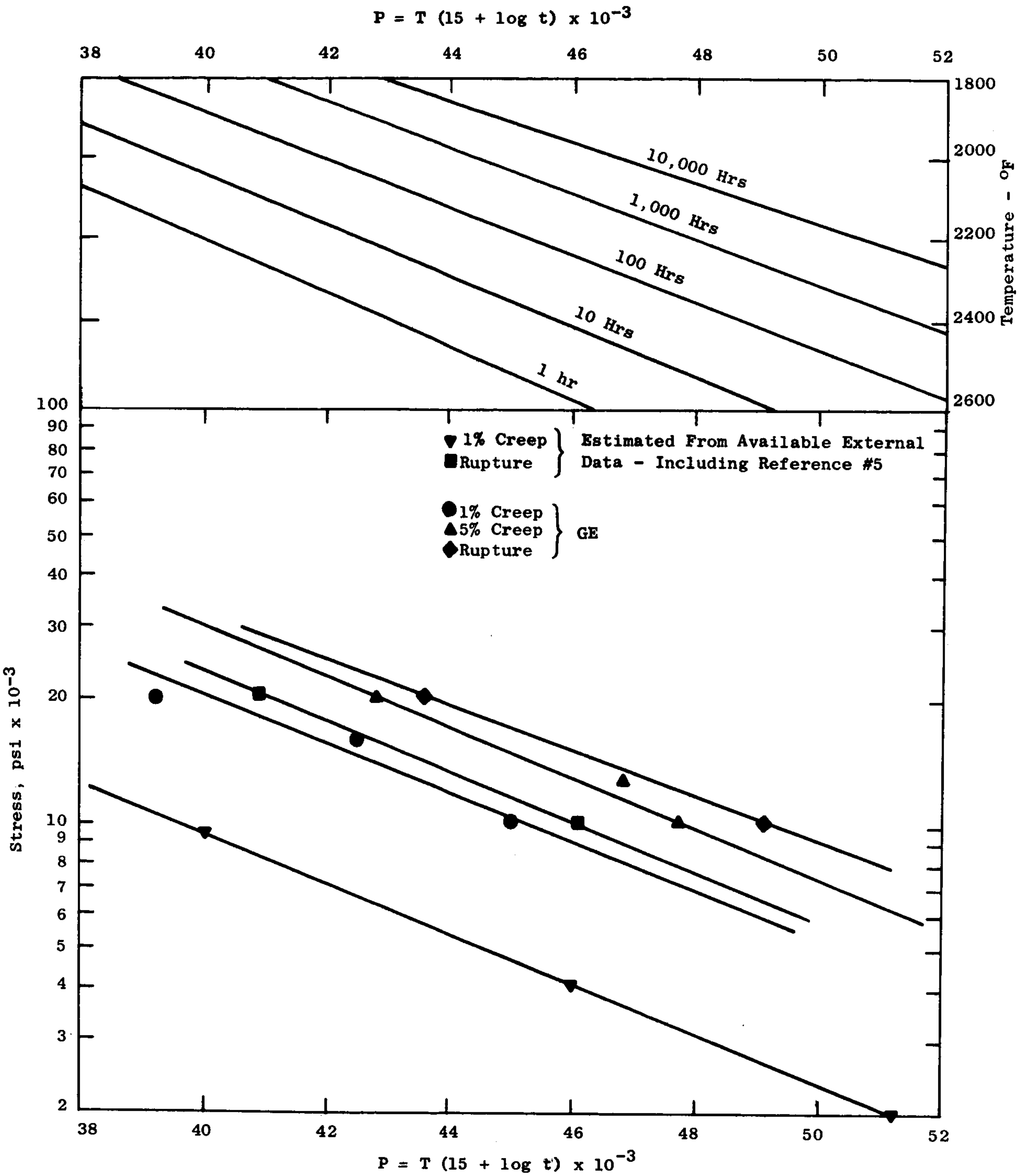


Figure 5. Creep-Rupture Properties of D-43 Alloy.

TABLE II

CHEMICAL ANALYSES OF D-43 ALLOY CREEP SPECIMENS

Element	Pre-test ppm	Post-test(1) ppm
Oxygen	48	110
Nitrogen	23	25
Hydrogen	5	1
Carbon	865	960

(1) 134 Hours at 2400°F in a Vacuum of 1×10^{-6} Torr (Baffled Oil Diffusion Pump System).

Using the test data depicted in Figure 5, the temperature-time relationship to produce 5% strain in a D-43 alloy capsule, as a result of the potassium vapor pressure, can be determined; the calculated values are shown in Table III. On the basis of these results, the first D-43 alloy capsule will be placed on test at 2200°F. Temperature adjustments will be made during the initial portion of the test depending upon the observed creep rate, measured by alumina probes on the reduced wall section of the capsule, so as to attain approximately 5% strain in 1,000-2,000 hours. Although the desired strain-time relationship could be attained at lower temperatures by further reduction in the thickness of the capsule reduced wall section, problems associated with maintaining tolerances during machining and handling do not warrant this approach.

Three D-43 alloy capsules have been machined with a 0.020-inch thick reduced wall section. The partially recrystallized center core was removed by electrostatic machining. Although some of this material was used to machine the thermocouple wells for each capsule, the resulting structure variance between the capsule wall and the thermocouple wells should have no deleterious effect on the critical aspects of the test, i.e., the behavior of the material at the reduced wall section. The inside diameter was finished by honing to better than a 16 rms finish. Prior to machining the reduced wall sections, each capsule was tightly fitted onto an aluminum mandrel. The final machining was performed on a tracer lathe taking maximum cuts of 0.003 inch for the final 0.030 inch to minimize induced stresses. A photograph of an unassembled capsule is shown in Figure 6.

C. Test Facility

The designs for the heat shields, heaters, electrical terminals and capsule supporting components were completed, and subsequently reviewed and approved by the NASA Technical Manager. All machined parts have been received. Two remaining items, the high-temperature LVDT's and the high-purity alumina probes, are on order and they should be received by January 15, 1965. Manufacturing difficulties have postponed shipping of these items by over a month from the original date.

Severe "bumping" of potassium reflux corrosion capsules resulting from unstable boiling in the temperature range of 1400° to 1600°F has been observed in this laboratory and elsewhere (Ref. 4) and is a serious point of consideration in respect to this capsule test. Any deformation in the reduced 0.020-inch thick wall section and/or fracture of the alumina probes resulting from boiling instabilities of

TABLE III

TEMPERATURE-TIME RELATIONSHIPS TO PRODUCE 5% STRAIN IN A D-43 ALLOY CAPSULE WITH A 0.020-INCH THICK REDUCED WALL SECTION

Temperature °F	Potassium Pressure, psi	Effective Stress[(1)], psi	Time for 5% Strain, Hours
2000	152.3	4620	---
2050	175.5	5320	---
2100	201.0	6100	90,000
2150	228.2	6930	20,000
2200	259.4	7560	4,000
2250	292.0	8850	800
2300	326.0	9780	200

(1) $\sigma_e = \frac{\sqrt{3}}{2} \cdot \frac{Pr}{t}$ (Ref. 3)

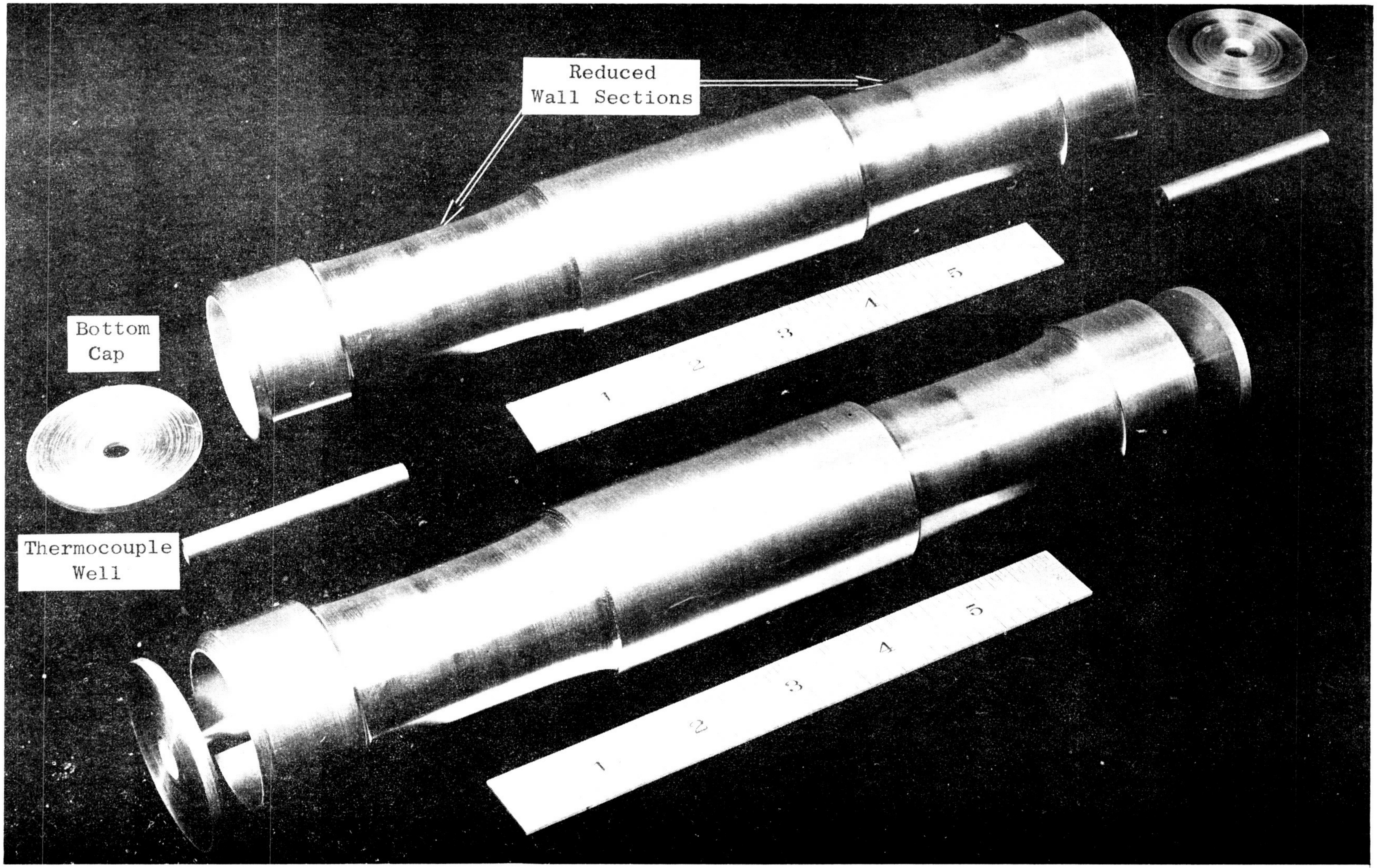


Figure 6. D-43 Reflux Corrosion Capsule with Reduced Wall Sections Machined in the Condensing and Liquid Regions. (C64123159) (C64123160)

this type during the initial heatup can not be tolerated. The use of a boiling nucleator has been shown to alleviate this problem (Ref. 4). The design to be used in this study is shown in Figure 7. Heating is supplied by a separately controlled tantalum wound resistance coil which is insulated by tantalum foil shielding. By maintaining a higher temperature in the nucleator than the capsule during the time the capsule is being heated through the critical temperature range, bubbles are nucleated at the root of the 1/16-inch diameter hole. This condition produces stable boiling of the potassium within the capsule itself. Once the critical temperature range is exceeded, the temperature in the nucleator will be maintained at the same level as the capsule.

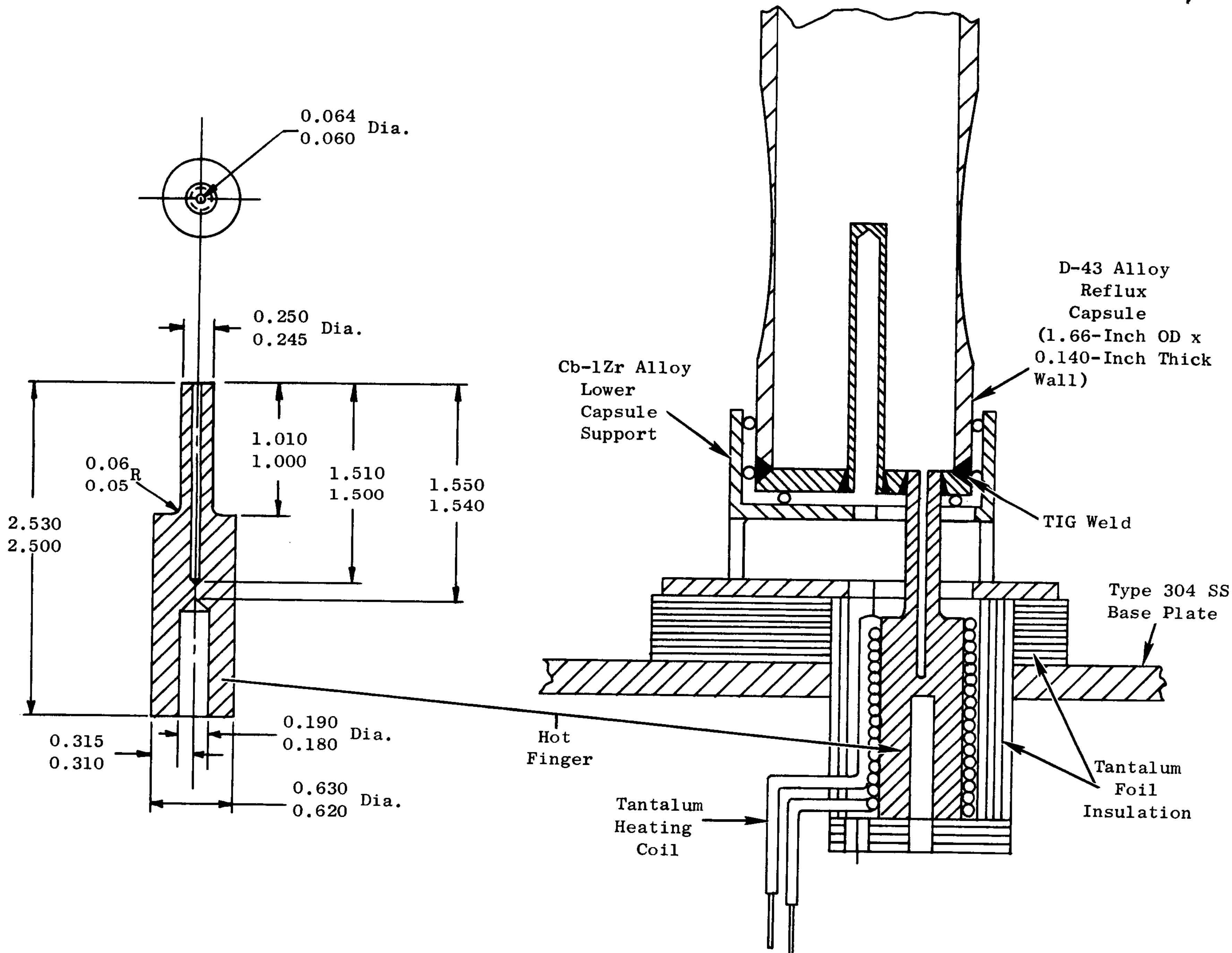


Figure 7. "Hot Finger" Design to Suppress Unstable Boiling of Potassium in D-43 Alloy Reflux Corrosion Capsule.

IV. TASK II - BIMETALLIC ISOTHERMAL CAPSULE TESTS

A. Materials Procurement

Chemical analyses (Ref. 3) of the 1-1/4-inch Schedule 80 stainless steel pipe to be used in capsule fabrication indicated the nitrogen content of the Type 316SS pipe (170 ppm) to be more than three times greater than the nitrogen content of the Type 321SS (52 ppm). Because of the possible difficulty in a comparative post-test evaluation of the nitrogen mass transfer effects of these two materials resulting from the different nitrogen contents, an additional heat of Type 321SS was purchased. Subsequent chemical analyses of this heat, Table IV, indicated a more amenable nitrogen analysis for comparable evaluation and for this reason the latter heat of Type 321SS was used in this study.

B. Capsule Fabrication

Two Type 321SS and two Type 316SS corrosion capsules, of the chemical analyses shown in Table IV, were fabricated to the design shown in Figure 8. A ballizing technique was employed to improve the finish of the inside diameter of the capsules. This technique consists of axially pressing a hardened (chrome-alloy tool steel) oversized ball (0.006-inch interference fit) through the pipe which has been well lubricated with a high-pressure wax. After removal of the wax with organic solvent and a light pickle in 20%HF, 20%HNO_3, 60%H_2O solution, the resulting finish was better than 16 rms. Metallographic examination of the internal diameter of the capsules indicated no deleterious effects are produced by employing this technique to reduce surface irregularities, Figures 9 and 10.

Bottom caps were tungsten-inert-gas welded to each capsule after ballizing; subsequent helium mass spectrometer leak tests indicated no leaks. Leak testing was performed to MIL-STD271C (Ships) specifications with a sensitivity of 5×10^{-10} std cc of air per second.

The surfaces of the 0.040-inch thick Cb-1Zr alloy sheet specimens, to be contained in the stainless steel capsules, were prepared by hand polishing through 640 grit paper, washing, light pickling in 20%HF, 20%HNO_3, and 60%H_2O solution and cleaning in ethanol. Room temperature tensile and 2000°F stress-rupture tests were conducted to establish the properties of the Cb-1Zr alloy in the pre-test condition. The results

TABLE IV

CHEMICAL ANALYSES OF 1-1/4-INCH DIAMETER

SCHEDULE 80 STAINLESS STEEL PIPE

Element	Type 316SS[1] ppm	Type 321SS[2] ppm
C	410	690
O	78	85
N	170	125
H	4	8
	w/o	w/o
Cr	17.39	17.44
Ni	12.33	11.90
Mn	1.86	1.62
Mo	2.24	--
Ti	--	0.46
Si	0.75	0.59
Cu	0.21	0.09

(1) Heat No. Carpenter 803776

(2) Heat No. B & W 27634X

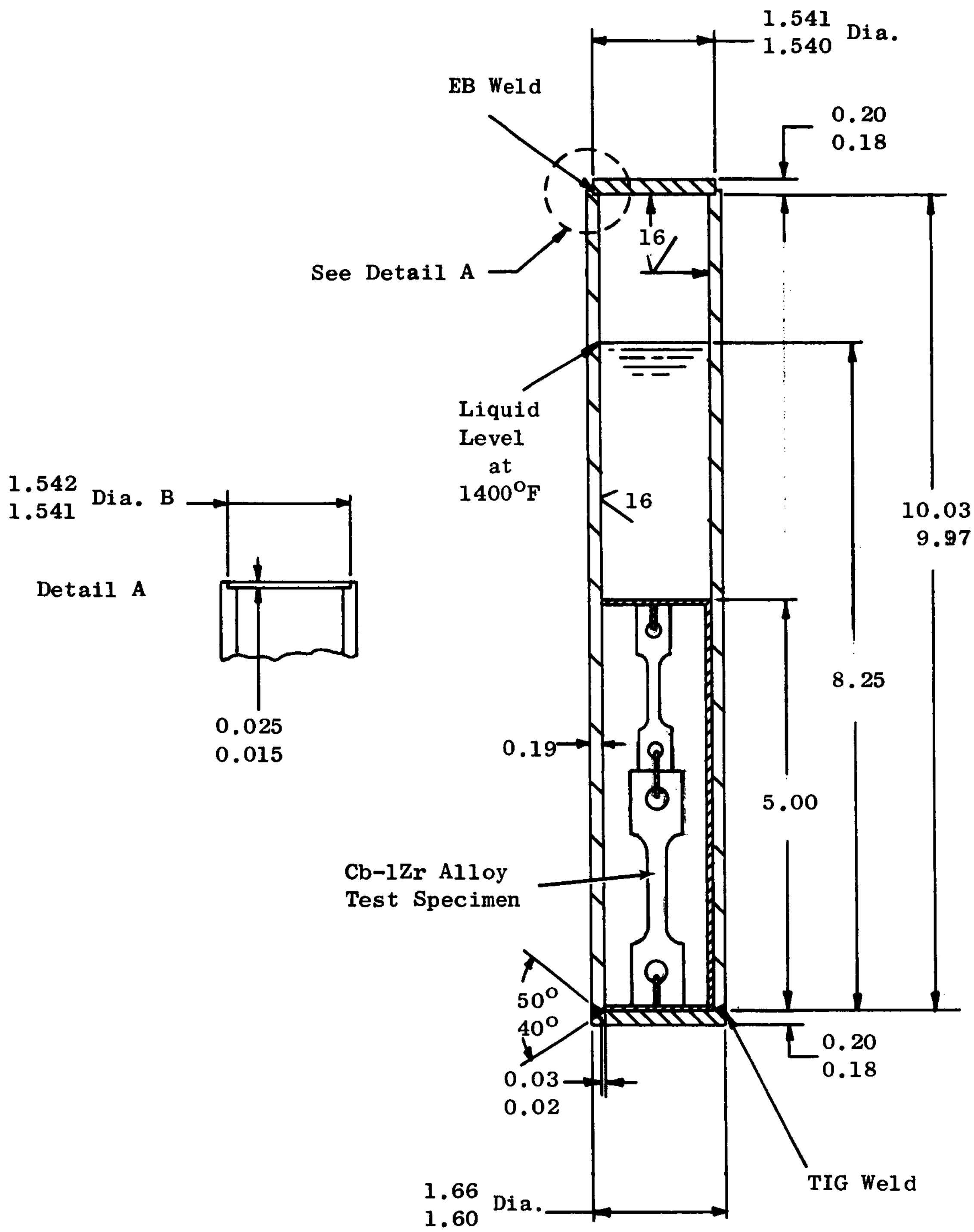


Figure 8. Stainless Steel Capsule Design.

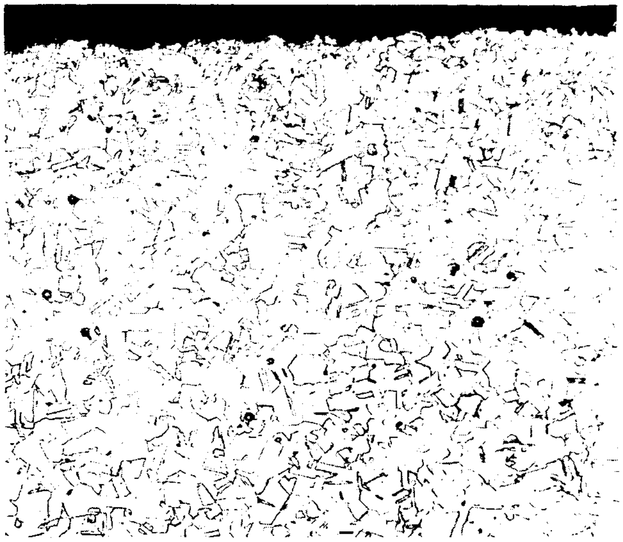


Figure 9. Transverse Section of As-Received Type 321SS Schedule 80 Pipe Showing Rough Internal Diameter Surface. (K5003)

Etchant: Aqua Regia Mag: 250X

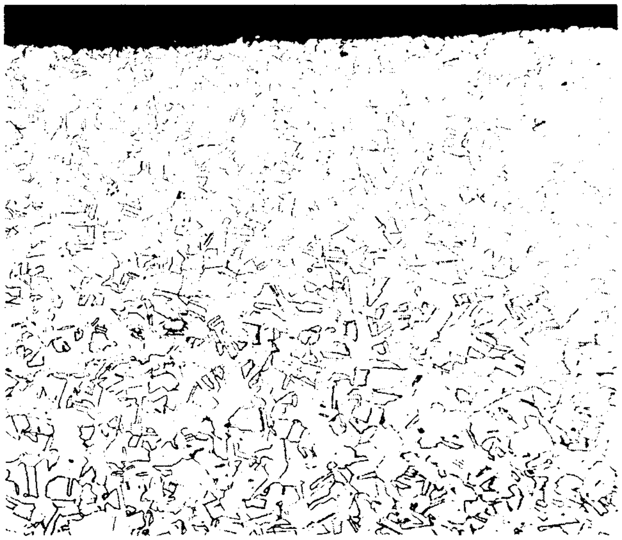


Figure 10. Transverse Section of Ballized Type 321SS Schedule 80 Pipe. Internal Diameter Surface Finished Better Than 16 rms with no Evidence of Flowed Metal. (K5004)

Etchant: Aqua Regia Mag: 250

are reported in Table V. A fixture to hold the Cb-1Zr alloy specimens vertical and equidistant from the internal walls of the capsules was constructed from 0.062-inch diameter Cb-1Zr alloy wire and is shown in Figure 11.

C. Capsule Filling

The four stainless steel capsules were filled with potassium and sealed under vacuum in a 30 KV electron beam welding chamber, Figure 12. The potassium used was transferred under argon pressure directly from the final hot trapping container (Ref. 3), Figure 13, into the evacuated chamber (3×10^{-5} torr) through a heated fill tube and into a calibrated stainless steel cup, Figure 14. The temperature of the cup was maintained at 200°F. Using the data depicted in Figure 15, calculations show that 135 cc of potassium at 200°F would result in a 8-inch fill height in the capsules at the test temperature of 1400°F. This height also is based on consideration of the volume displacement of the Cb-1Zr alloy specimens and fixture. The surface area ratio between the stainless steel and the Cb-1Zr alloy in contact with liquid potassium has been shown to be critical (Ref. 2). The relationships described in this study constitute a 4:1 ratio which is more than sufficient to observe mass transfer of nitrogen and carbon in Type 316SS at temperatures near 1500°F (Ref. 2).

The measured potassium was poured from the cup through a heated stainless steel funnel into the capsule. Each capsule was positioned under the funnel by means of a flexible cable which was attached on one end to a gear train on the table and on the other end to a crank outside the chamber, Figures 12 and 16. When filled, each capsule was rotated under a manipulator which was used to position the capsule cap in the machined, slotted well on the capsule top. The manipulator is sealed on the outside of the chamber by a bellows. The capsule was positioned under the EB gun by means of a flexible cable in conjunction with the table drive. The gear on each capsule was thus adjusted to mesh with the welding drive gear such that the capsules rotated around its axis during welding. This gear is controlled by a variable speed motor which can be adjusted to obtain the optimum welding speed. Copper chill blocks were fitted on each capsule to reduce the heat conduction along the capsule during welding and the resulting possible vaporization of the potassium causing unsound welds.

TABLE V

PRE-TEST STRENGTH ANALYSIS OF MCN-454 Cb-1Zr ALLOY 0.040-INCH SHEET

Tensile Properties

Temp. °F	Ultimate Tensile Strength, psi	Yield Strength .2% Offset, psi	Elongation, %
RT	39,900	23,300	37.0
RT	38,900	21,900	40.4

Stress-Rupture(1) Properties

Temp. °F	Stress, psi	Rupture Time Hours	Elongation, %
2000	10,000	252	44
2000	8,000	164(2)	3.4

(1) Tests Conducted in Vacuum; 1×10^{-7} Torr. (Getter-Ion Pump System)

(2) Did Not Rupture; Test Discontinued.

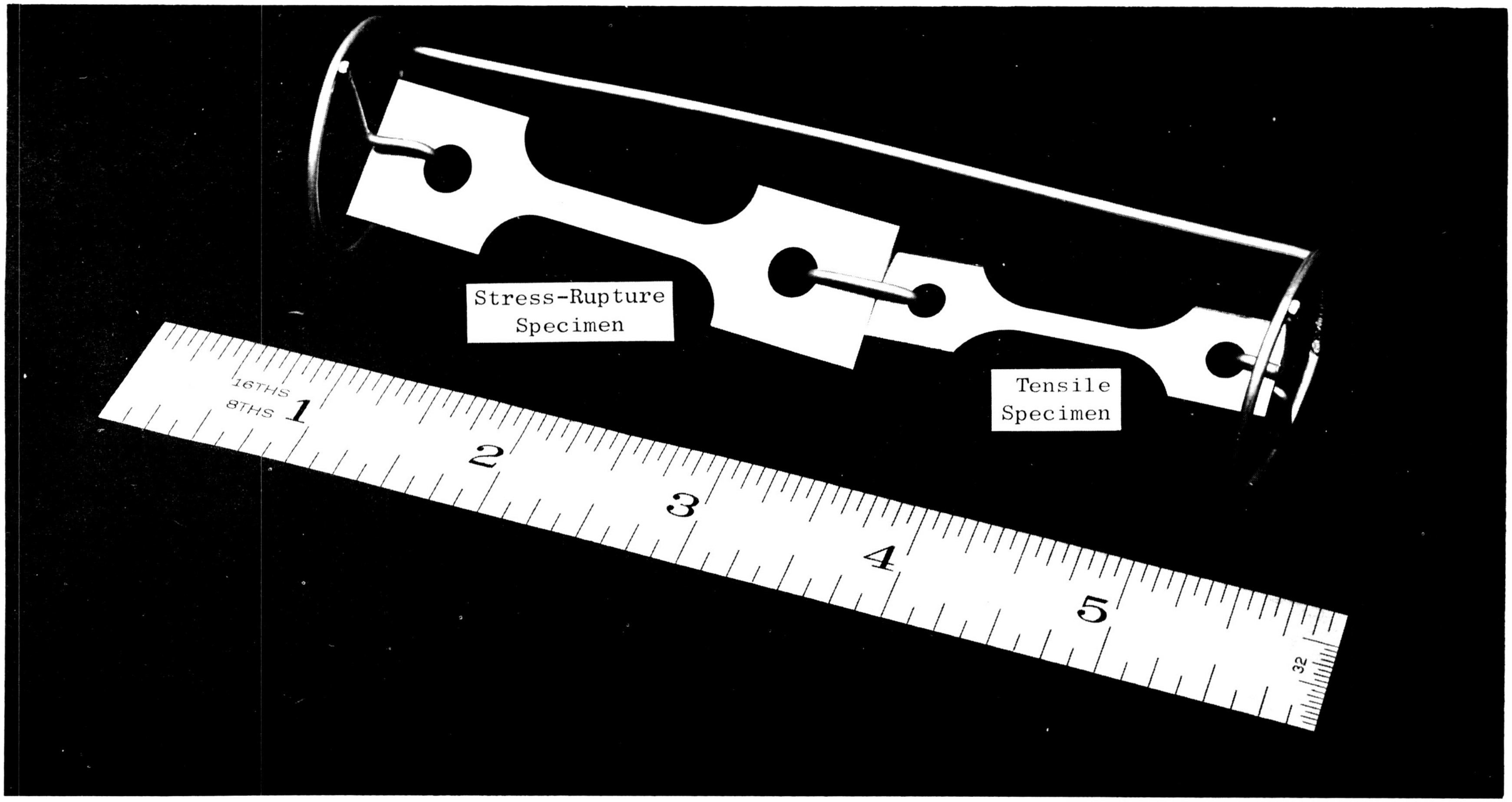


Figure 11. Cb-1Zr Alloy Holding Fixture for Positioning Cb-1Zr Alloy Test Specimens Within Stainless Steel Corrosion Capsules. (C64120206)

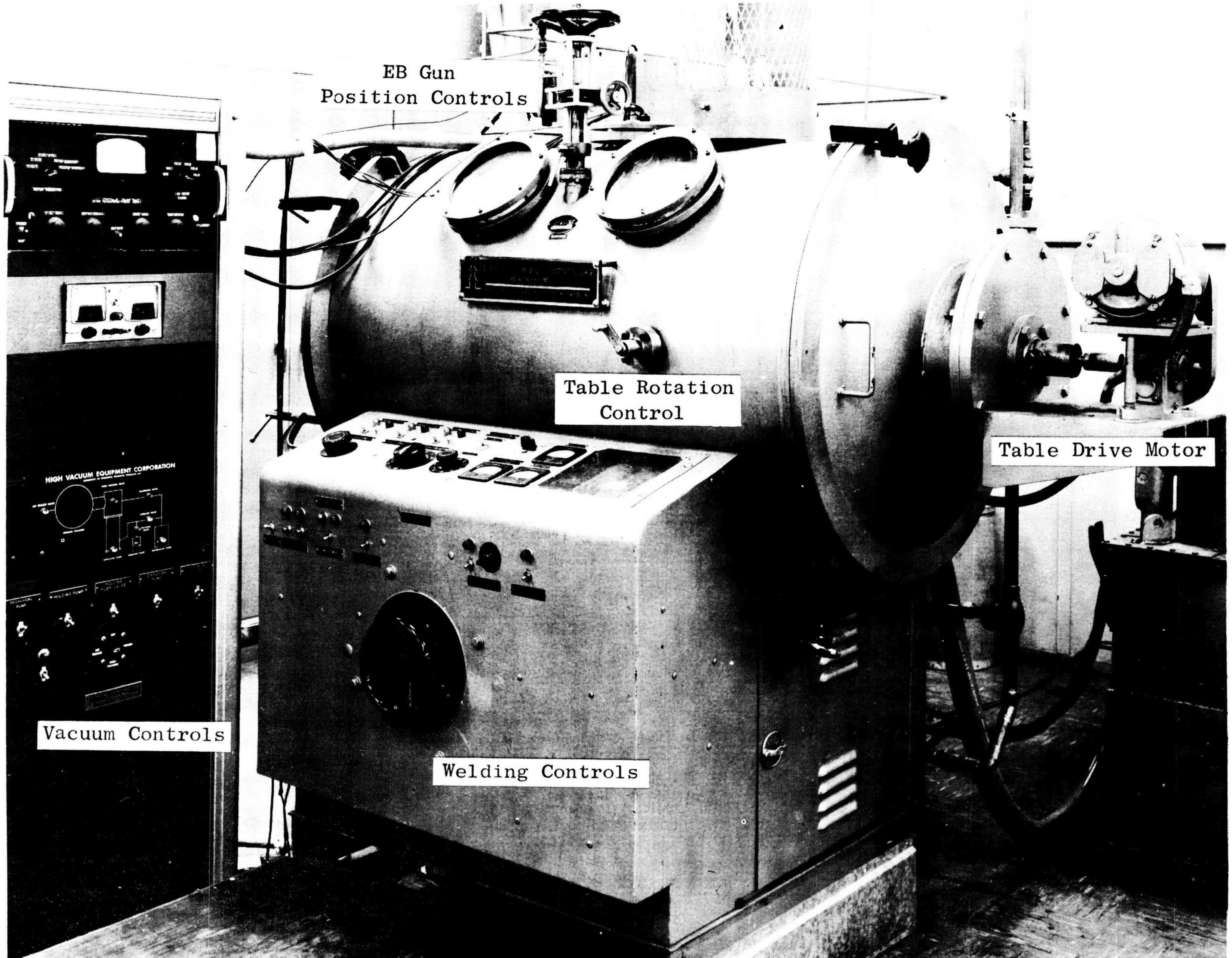


Figure 12. 30KV Electron Beam Welding Chamber and Controls Used to Fill Corrosion Capsules with Potassium. (C64121619)

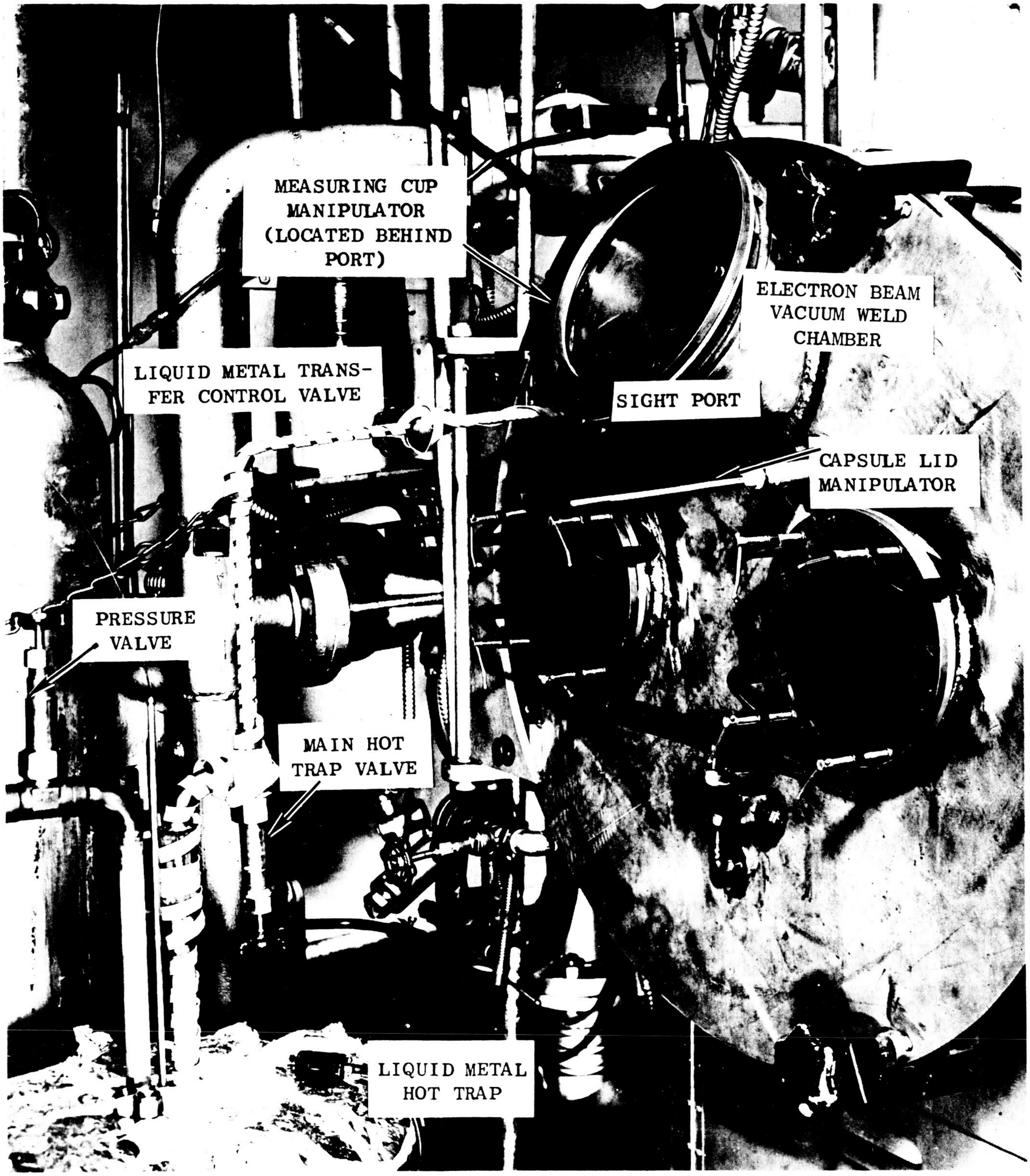


Figure 13. External View of the Facility for Potassium Transfer; the Hot Trap is Shown Attached to the Vacuum Tank Closure. (C63041623)

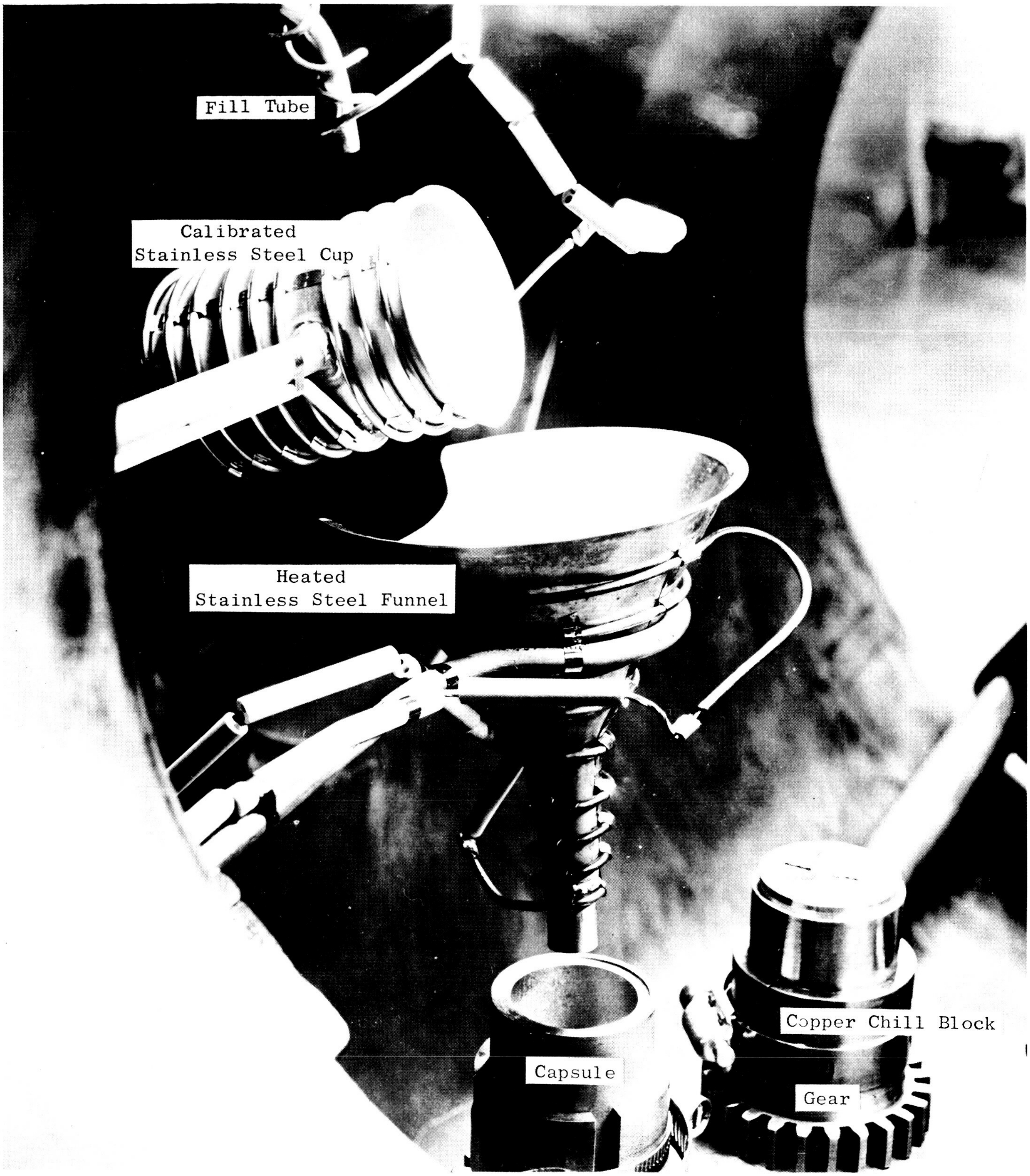


Figure 14. Internal Arrangement of Facility for Filling Corrosion Capsules with Potassium as Viewed Through Sight Port of Electron Beam Welding Chamber. (C64121621)

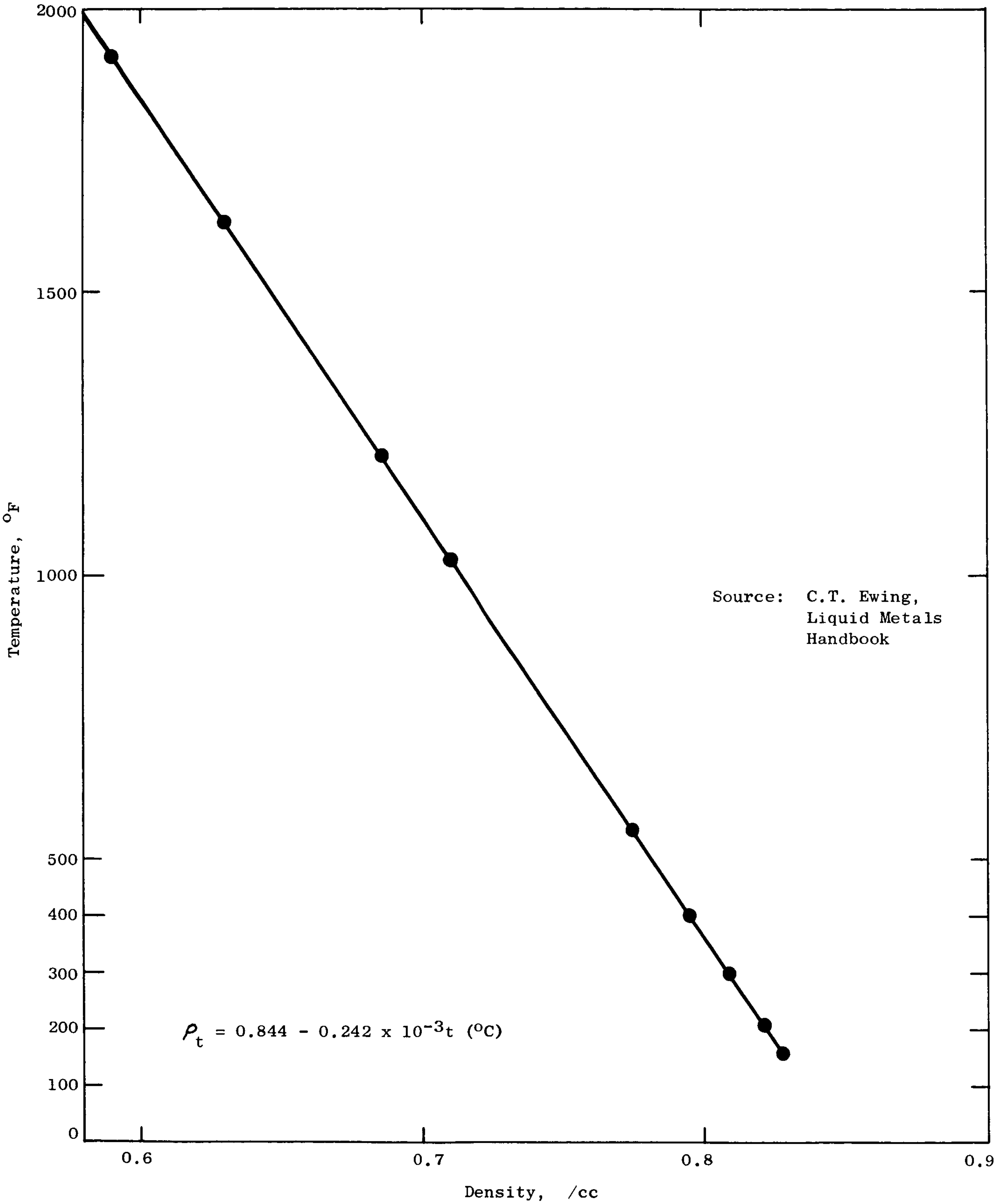


Figure 15. Density of Liquid Potassium as a Function of Temperature.

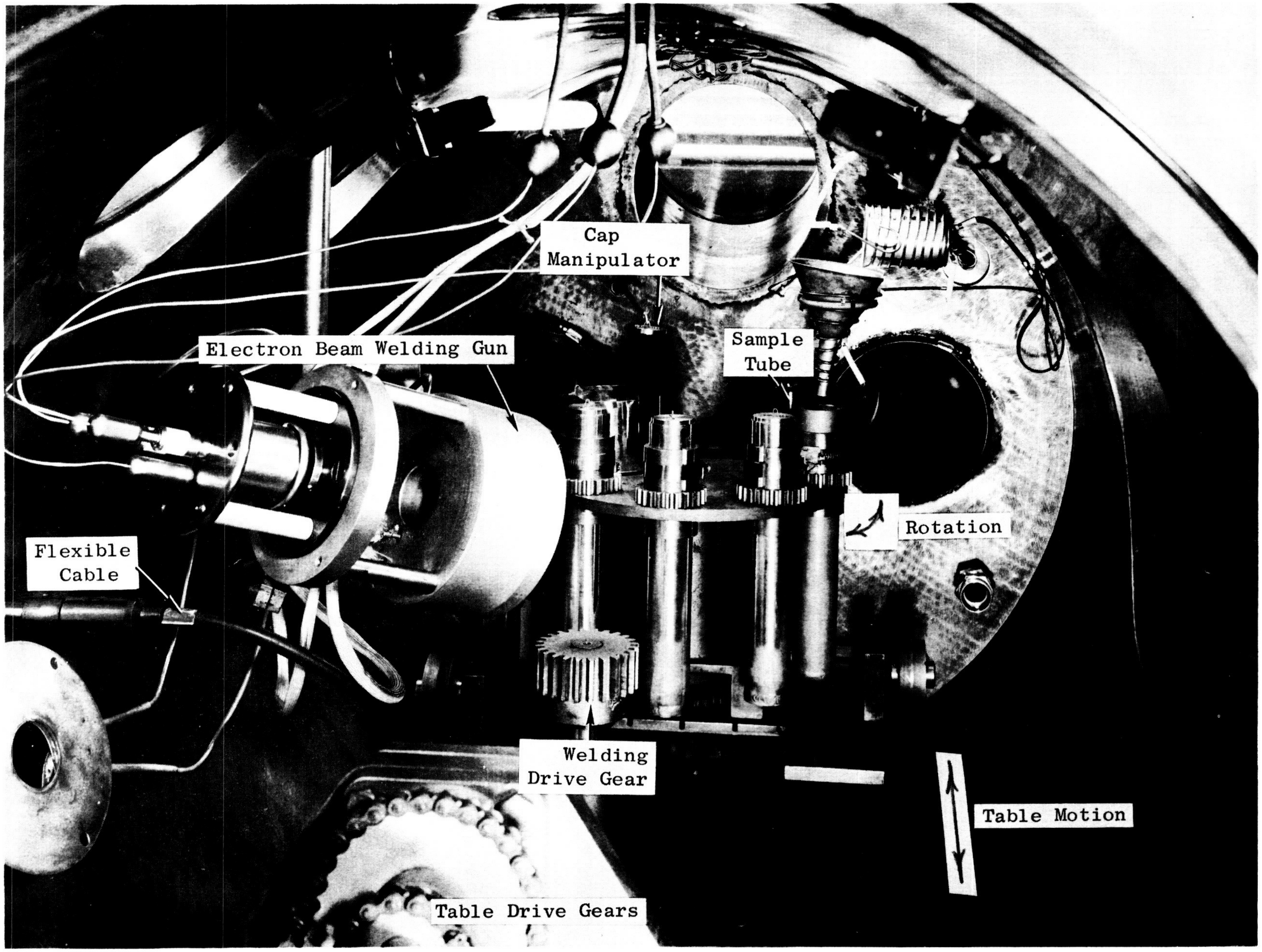


Figure 16. Internal View of Facility Showing the Components Required to Fill Capsules with Potassium. (C64121620)

Samples of the potassium used to fill the capsules were taken in two locations. One sample was obtained by filling a stainless steel tube in the same manner as the capsules are filled, Figure 14. The other sample was taken by removing a section of the fill line between the chamber and the hot trap. The resulting oxygen analyses by the mercury amalgamation method showed 2.5 ppm oxygen in the potassium taken from the chamber sample and 3 ppm oxygen in the potassium taken from the fill tube. Metallic impurities are being determined by spectrographic analysis. A can was placed on the rotating table to provide means of dumping any excess potassium during filling. The first cup of potassium was discarded in this manner as a result of possible contamination from pickup along the walls of the empty fill line.

The four filled and sealed capsules were examined radiographically to assure sound electron beam welds and subsequently put in test.

D. Test Facility

The air environment furnace to be used for testing the stainless steel isothermal corrosion capsules, Figure 17, was designed and constructed during the report interim. The facility consists of four 1340 watt, nichrome-wound resistance elements, individually controlled with General Electric Volt-Pac 9H60 variable transformers. Each capsule is held in place with an adjustable tie bolt attached to a stainless steel retaining plate. The 8-point recorder allows dual temperature monitoring of each capsule with chromel-alumel thermocouples.

E. Capsule Testing

The 1,000-hour bimetallic corrosion capsule test at 1400°F was initiated on December 18, 1964. Two beaded chromel-alumel thermocouples were spot welded to the sides of two capsules at the mid-point on the longitudinal axis and 180° apart. One thermocouple was attached to each of the remaining two capsules in the same location and the second thermocouple was attached near the top of one of the capsules and near the bottom of the other capsule. Temperature measurement to date has indicated a maximum of 15°F drift; however, temperature variance from capsule to capsule at the mid-point is less than 10°F. A maximum of 20°F temperature differential was measured between the top and bottom thermocouples.

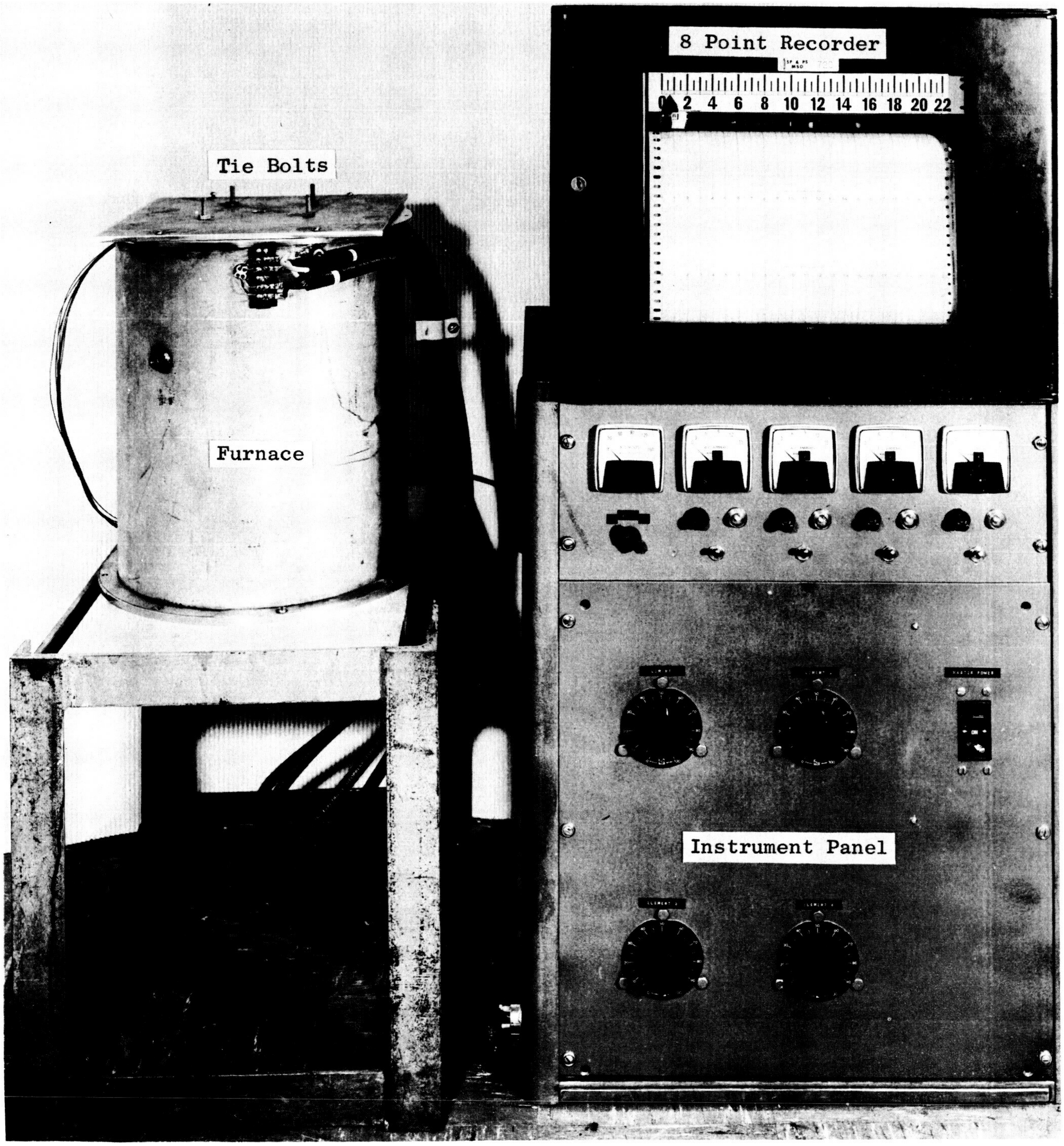


Figure 17. Test Facility for Isothermal Corrosion Capsule Tests.
(C64121039)

V. FUTURE PLANS

A. Task I

1. Complete assembly of test facility components and check-out instrumentation.

2. Fabricate and fill the first D-43 alloy capsule.

3. Initiate the first capsule test at 2200°F.

B. Task II

1. Complete the 1,000-hour capsule test at 1400°F and initiate post-test evaluation.

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(4) Hoffman, E. E., "Boiling Potassium Stability Studies," Metals and Ceramics Division Annual Progress Report, May 3, 1963, ORNL-3470, p 114-118.

(5) Hall, R. W., Titran, R. H., "Creep Properties of Columbium Alloys in Very High Vacuum," Lewis Technical Preprint 15-63.

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